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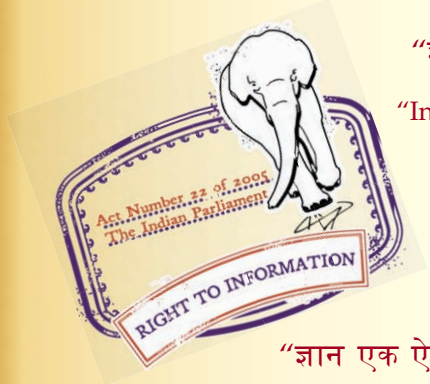
IS 8941 (1997): Chrysazin-specification [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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(Reaffirmed 2002)

भारतीय मानक

क्रिसाजिन — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

CHRYSAZIN — SPECIFICATION

(First Revision)

ICS 71.080.80

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

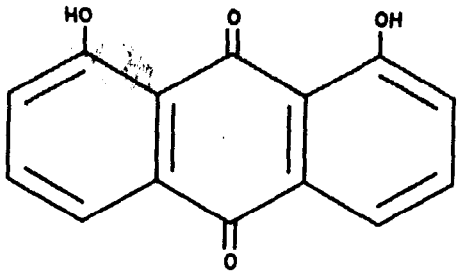
July 1997

Price Group 3

FOREWORD

This Indian Standards (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Chrysazin ($C_{14}H_8O_4$), chemically described as 1, 8-dihydroxy- anthraquinone is an important dye intermediate used in the manufacture of disperse dyes. It is represented by the following structural formula:



CHRYSAZIN
(MOLECULAR MASS 240.2)
CAS REGISTRY No. [117-10-2]

This standard was first published in 1978. The committee responsible for the preparation of this standard decided to update it in the light of experience gained. In this version, the requirement for impurities such as 1, 5 Dihydroxyanthraquinone and 1-Hydroxyanthraquinone have been stipulated. An alternate High Pressure Liquid Chromatography (HPLC) method for the determination of 1,8-Dihydroxyanthraquinone content and other impurities has also been incorporated in this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHRYSAZIN — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Chrysazin.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the edition indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests of dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a bright yellow powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material,

b) Indication of the source of manufacture,

c) Net mass of the material,

d) Batch number, and

e) Month and year of the manufacture.

4.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provision of *Bureau of Indian Standards Act*, 1986 and Rules and Regulations made thereunder. The details of conditions under which the licence for

Table 1 Requirements for Chrysazin

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristics	Requirement	Method of Test Ref to
(1)	(2)	(3)	(4)
i)	1,8-Dihydroxyanthraquinone, percent by mass, *Min*	88.0	Annex A
ii)	1, 5-Dihydroxyanthraquinone, percent by mass, *Max*	2.0	Annex A
iii)	1-Hydroxyanthraquinone, percent by mass, *Max*	1.0	Annex A
iv)	Melting Point	Shall melt within the range of 3°C between limits 191 to 195°C	Cl 8 of IS 5299
v)	Moisture, percent by mass, *Max*	0.5	Cl 9.3 of IS 5299
vi)	Sulphated ash, percent by mass, *Max*	2.0	Cl 11.2 of IS 5299

the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299.

5.2 Number of Tests

5.2.1 Test for the determination of 1,8-dihydroxy-anthraquinone, 1, 5-dihydroxyanthraquinone and 1-Hydroxyanthraquinone shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, melting point, moisture and sulphated ash shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of 1,8-dihydroxyanthraquinone, 1,5-dihydroxyanthraquinone and 1-Hydroxyanthraquinone if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods referred in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i), (ii) *and* (iii)]

DETERMINATION OF 1, 8-DIHYDROXYANTHRAQUINONE AND ITS IMPURITIES

A-1 GENERAL

Two methods, namely, Method A 'Column chromatography' and Method B 'High pressure liquid chromatography (HPLC)' have been prescribed for the determination of 1,8-Dihydroxyanthraquinone and its impurities. Any of these methods can be used on routine basis. However in the event of any dispute, HPLC method shall be treated as a referee method.

A-2 METHOD A

A-2.1 Outline of the Method

The purity of the material is estimated by the chromatographic method. 1 : 8-Dihydroxyan-thraquinone is separated by chromatography and determined quantitatively by spectrophotometer.

A-2.2 Reagents

A-2.2.1 *Sodium Carbonate*

Sodium carbonate of the following specifications shall be used as it is found to be suitable for chromatographic analysis to obtain effective separation:

Fineness	*Percentage*
Retained on 150-micron IS sieve	0.0
Retained on 75-micron IS sieve	30 to 45
Passing through 53-micron IS sieve	45 to 50

A-2.2.1.1 *Activation*

Sodium carbonate of the above fineness should be activated before use by drying overnight at 200°C.

A-2.2.2 *Acetone*

Pure, dried over fused calcium chloride, filtered and distilled before use. Determine its water content by the Karl Fischer method.

A-2.2.3 *Solvent Mixture (A)*

Pure distilled acetone adjusted to contain 1.0 percent water (m/v).

A-2.2.4 *Solvent Mixture (B)*

Pure distilled acetone adjusted to contain 2.0 percent water (m/v).

A-2.2.5 *Solvent Mixture (C)*

Pure distilled acetone adjusted to contain 3.0 percent water (m/v).

A-2.2.6 *Chromatographically Pure 1,8-Dihydroxyanthraquinone*

Dissolve 200 mg of 1,8-dihydroxyanthraquinone in 200 ml of acetone. Boil and reflux for 15 minutes. Cool to room temperature and transfer to 250-ml volumetric flask and make up to the mark with acetone. Mix well and call it solution (X). Transfer the solution (X) gradually to the chromatographic column. When all the solution loaded has entered the column and the glass-wool at the top of the column is just dry, add small quantities of the solvent mixture (A) without disturbing the surface until the bands start moving down the column. Continue elution with solvent mixture (A) until there is distinct separation of bands which will be in the following order (from bottom to top):

a) Small yellow band of 1-hydroxyanthraquinone
b) Small pink band of 1,5-dihydroxyanthraquinone
c) Main pink band of 1,8-dihydroxyanthraquinone, and
d) Strongly absorbed purple band at the top.

Elute the small yellow band of 1-hydroxyanthraquinone with solvent mixture (A). After the small yellow band is completely eluted, change the eluent to solvent mixture (B) when the next small pink band of 1,5-dihydroxyanthraquinone will start moving dowr the column. After this band is completely eluted change the eluent to solvent mixture (C). The main pink band of 1,8-dihydroxynathraquinone will start moving down the column. A battery of five to six tubes is kept in operation for collecting a sizable quantity of the material. Filter through filter paper to remove any insoluble. Concentrate the elute of 1,8-dihydroxyanthraquinone by distillation until crystallization occurs on cooling to room temperature. Filter on a sintered glass crucible of porosity G4 and wash first with hot water and then with cold water. Suck dry and finally dry in a vacuum oven at 100°C.

In order to check its purity, dissolve about 10 mg (accurately weighed) in 10 ml of acetone and pass the cooled solution through a chromatographic column eluting with solvent mixture (C) as necessary. Collect the band carefully (only one band should appear) and determine its optical density at 428 nm. Make up a solution of purified crystals using the same proportions of crystals to solvent. Determine the optical density in the same way. A difference in the two figures for optical density amounting to more than 0.003 indicates the presence of an impurity. The chromatographic purification shall in such a case be repeated until this check test is satisfied.

A-2.2.7 *1-Hydroxyanthraquinone*

Pure (100 percent on dry basis).

A-2.2.8 *1,5-Dihydroxyanthraquinone*

Pure (100 percent on dry basis).

A-2.2.9 *Glacial Acetic Acid*

A-2.3 Apparatus

A-2.3.1 *Chromatographic Column*

A glass tube 40 cm long and having 1.5 cm as its internal diameter, joined with a 50-ml thistle funnel at the upper end and fitted with a stopcock at the lower end, is set-up vertically, such that the percolation passing through the tube can be collected conveniently in a 250-ml conical flask. Place a glass-wool plug in the tube and press it to the bottom of the tube by means of a glass rod flattened latitudinally at the end. Place a disc of filter paper cut to the approximate internal diameter of the chromatographic tube on top of the glass-wool.

Prepare a slurry of sodium carbonate in solvent mixture (A) and pour it into the chromatographic tube. Wash down the sides of the tube and pack the column by lightly tapping the tube. Place first a disc of filter paper and then a glass-wool plug at the top of the column. Care should be taken to see that at least a 2-cm layer of the solvent remains always on top of the column. On no account shall the column be left dry. In the event of the latter happening, reslurry the sodium carbonate in the tube and repack.

A-2.3.2 *Vacuum Oven*

A-2.3.3 *Spectrophotometer*

Capable of taking reading at 407 nm and 428 nm.

A-2.4 Standard Calibration Graph

A-2.4.1 Prepare a number of standard solutions of the chromatographically pure 1,8-dihydroxyanthraquinone in solvent mixture (C) varying in concentrations from 0.6 to 2.0 mg/100 ml with a difference of 0.2 mg/100 ml between two successive concentrations. Before making up the volume to 100 ml add 4 ml of glacial acetic acid in each flask.

A-2.4.2 Prepare a calibration graph of 1-hydroxyanthraquinone in an identical way as **A-2.4.1** using solvent mixture (A).

A-2.4.3 Prepare a calibration graph of 1,5-dihydroxyanthraquinone in an identical way as **A-2.4.1** using solvent mixture (B).

A-2.4.4 Take readings for optical density or percentage transmittance for the solutions of various concentrations (*see* **A-2.4.1**) at a wavelength of 428 nm (*see* Notes) using specifically matched cells specified for the particular spectrophotometer used. The temperature of the solution immediately before and after measurements shall be 27±2°C.

Take readings for optical density or percentage transmittance for the solutions of various concentrations (*see* **A-2.4.2**) at a wavelength of 407 nm (*see* Notes) using specially matched cells as above.

Also take readings for optical density or percentage transmittance for the solutions of various concentrations (*see* **A-2.4.3**) at a wavelength of 428 nm (*see* Notes) using specially matched cells as above.

NOTES

1 Maximum absorption (or minimum transmittance) has been at a wavelength of 407 nm and 428 nm by standard instruments. However, this may need checking with the particular instrument to be used. Several readings for absorbance using solutions of varying concentrations are taken at different wavelengths and that for maximum absorption determined from the graph.

2 The standard calibration curve and the spectrophotometer shall be checked for accuracy from time to time.

A-2.4.5 When the instrument is calibrated in percentage transmittance only, the optical density may be read out from a standard table. Plot the calibration curve of concentration (in mg/100 ml) against optical density for 1,8-dihydroxy anthraquinone, 1-hydroxyanthraquinone and 1,5-dihydroxy anthraquinone.

A-2.5 Procedure

A-2.5.1 Weigh accurately about 50 mg of the dry pulverized sample of the material in a 250-ml conical flask. Add about 200 ml of distilled, dry acetone and reflux for about 15 minutes (using a condenser) to effect complete solution. Cool to room temperature by keeping in water bath. Transfer the solution to a dry 250-ml volumetric flask. Rinse the conical flask with small amounts of acetone and transfer the rinsing also to the volumetric flask. Make up to the mark with acetone and mix the solution (1) well.

A-2.5.2 Prepare a solvent-soaked adsorption column of sodium carbonate using a chromatographic tube 40 cm in length and 1.5 cm internal diameter. Standardized sodium carbonate suitable for chromatographic analysis is used as adsorbent and solvent mixture (A) as the solvent. Pack the column (*see* **A-2.3.1**).

A-2.5.3 Load 10 ml of solution (1) of 1,8-dihydroxyanthraquinone. When all the solution has entered the column and glass-wool at the top of the column is just dry, add a small quantity of solvent mixture (A). Go on adding the solvent in small lots until the glass-wool on the top of the column is colourless and all the coloured solution is washed into the column. Now add more solvent mixture (A) carefully without disturbing the surface of the column and continue further addition of solvent mixture (A). Collect the small yellow band of 1-hydroxyanthraquinone in 100-ml volumetric flask containing 4.0 ml of glacial acetic acid. Make up to mark with solvent mixture (A). Mix well. Call this solution (2).

A-2.5.4 Now change the eluent to solvent mixture (B), the small pink band of 1,5-dihydroxyanthraquinone will start moving down the column. Collect the band of 1,5-dihydroxyanthraquinone in 100-ml volumetric flask containing 4.0 ml of glacial acetic acid. Make up to mark with solvent mixture (B). Mix well. Call this solution (3).

Change the eluent to solvent mixture (C). The main pink band of 1,8-dihydroxyanthraquinone will start moving down the column. Collect the main band of 1,8-dihydroxyanthraquinone in 100-ml volumetric flask containing 4.0 ml of glacial acetic acid. Make up to mark with solvent mixture (C). Mix well. Call this solution (4).

Adjust the wavelength of maximum absorption at 407 nm for solution (2) and 428 nm for solutions (3) and (4). Adjust the instrument in such a way that the transmittance through the blank solution is recorded at 100 percent reading after inserting the cell with the blank solution (solvent mixtures A, B and C, respectively). Now replace the cell with solution (2), (3) and (4) of the sample and read the percentage transmittance. Referring to the standard tables for conversion of transmittance to optical density, read out concentration C against optical density from standard calibration graph in terms of mg/100 ml of the final dilute solutions (2), (3) and (4).

A-2.6 Calculation

a) 1-Hydroxyanthraquinone, percent by mass $= \dfrac{A \times 2\,500}{M}$

where

A = Mass in mg of 1-hydroxyanthraquinone contained in 100 ml of solution (2).

M = Mass in mg of the material under test.

b) 1,5-Dihydroxyanthraquinone, percent by mass $= \frac{B \times 2\,500}{M}$

where

B = Mass in mg of 1,5-dihydroxyanthraquinone contained in 100 ml of solution (3).

M = Mass in mg of the material under test.

c) 1,8-Dihydroxyanthraquinone, percent by mass $= \frac{C \times 2\,500}{M}$

where

C = Mass in mg of 1,8-dihydroxyanthraquinone contained in 100 ml of solution (4).

M = Mass in mg of the material under test.

A-3 METHOD B

A-3.1 Outline of the Method

A-3.1.1 Assay and impurities of 1,8-Dihydroxyanthraquinone are estimated by High Pressure Liquid Chromatographic method.

A-3.2 Reagents

A-3.2.1 *1,8-Dihydroxyanthraquinone*

Pure (100 percent on dry basis).

A-3.2.2 *1,5-Dihydroxyanthraquinone*

Pure (100 percent on dry basis).

A-3.2.3 *1-Hydroxyanthraquinone*

Pure (100 percent on dry basis).

A-3.2.4 *Tetrahydrofuran*

Spectroscopy grade.

A-3.2.5 *Acetic acid*

Extra pure.

A-3.2.6 *Distilled water*

A-3.3 Apparatus

A-3.3.1 *High Pressure Liquid Chromatograph*

Any suitable chromatograph with a reverse phase column. Typical parameters of Waters HPLC is given below:

Column	—	u-Bondapak C_{18}
Temperature	—	25°C
Wavelength	—	254 nm
Flow rate	—	1.2 ml/Min.
Attenuator	—	0.1
Chart speed	—	10 mm/Min.
Load on column	—	5.0 µl
Mobile phase	—	

$$\frac{\text{Tetrahydrofuran}}{50} : \frac{\text{Distilled water}}{50} : \frac{\text{Acetic acid}}{1.5}$$

A-3.3.2 *'A' Grade Volumetric Flask*

A-3.4 Procedure

A-3.4.1 *Preparation of Sample Solution*

A-3.4.1.1 Weigh accurately 0.05 gm of well mixed (previously ground and dried) sample and transfer to 100 ml standard volumetric flask using 80 ml of Tetrahydrofuran (**A-3.2.4**). Dissolve it in hot water bath. Cool to room temperature. Dilute to mark using Tetrahydrofuran and mix well. Call this solution 'S'.

Pipette 10 ml of solution 'S' in 100 ml standard volumetric flask and dilute to mark using Tetrahydrofuran. Mix well. Call this solution 'S_1'.

A-3.4.2 *Preparation of Standard Solution*

A-3.4.2.1 *1,8-Dihydroxyanthraquinone*, Pure. Weigh accurately 0.05 g of pure 1,8-dihydroxyan-thraquinone (previously dried) and transfer to 100 ml standard volumetric flask using 80 ml of tetrahydrofuran (**A-3.2.4**). Dissolve it in hot water bath. Cool to room temperature. Dilute to mark using tetrahydrofuran and mix well. Call this solution 'A'.

Pipette 10 ml of solution 'A' in 100 ml standard volumetric flask and dilute to mark using tetrahydrofuran. Mix well. Call this solution 'A_1'.

A-3.4.2.2 *1,5-Dihydroxyanthraquinone*, Pure. Weigh accurately 0.05 g of pure 1,5-dihydroxyan-thraquinone (previously dried) and transfer to 100 ml standard volumetric flask using 80 ml of tetrahydrofuran. Prepare a solution 'B_1' in an identical way as prepared in **A-3.4.2.1**.

A-3.4.2.3 *1-Hydroxyanthraquinone*, Pure. Weigh accurately 0.05 g of pure 1-hydroxyan-thraquinone (previously dried) and transfer to 100 ml standard volumetric flask using 80 ml of tetrahydrofuran. Prepare a solution 'C' and 'C_1' in an identical way as prepared in **A-3.4.2.1.**

A-3.4.3 *Determination of 1,8-Dihydroxyanthraquinone and Impurities as 1,5-Dihydroxyanthraquinone and 1-Hydroxyanthraquinone*

A-3.4.3.1 When steady base line is obtained, inject 5.0 µl of each standard solution A_1, B_1 and C_1. Also inject 5.0 µl of sample solution S_1.

Measure height and base width of the peak and

calculate. A typical chromatogram is shown in Fig 1.

A-3.4.3.2 *Calculation*

1,8-Dihydroxyanthraquinone, percent by mass

$$= \frac{\text{Concentration of standard} \times \text{Area of peak in the sample} \times \text{Strength of standard}}{\text{Concentration of sample} \times \text{Area of peak of standard}}$$

1,5-Dihydroxyanthraquinone, percent by mass

= Same as above

1-Hydroxyanthraquinone, percent by mass

= Same as above

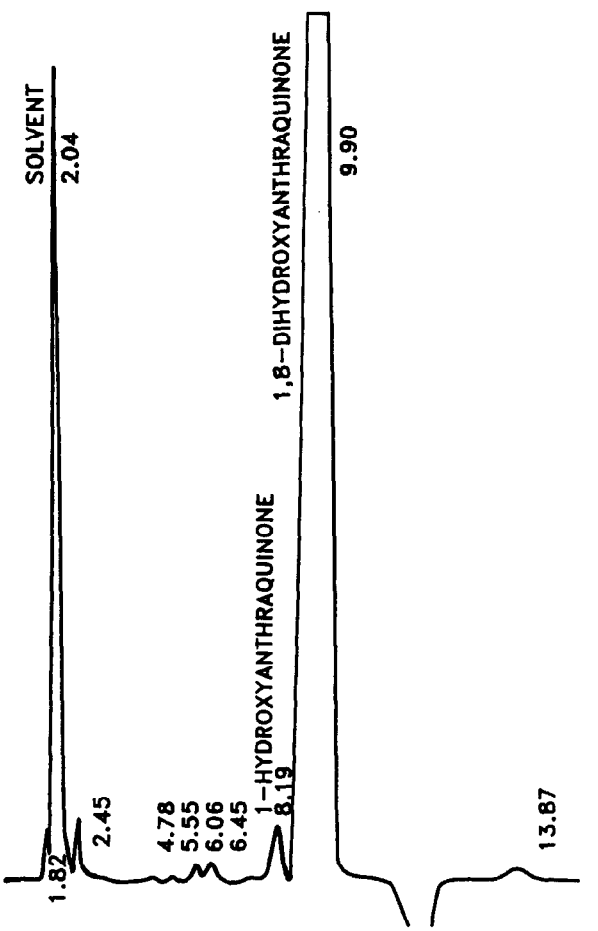


FIG. 1 A TYPICAL HIGH PRESSURE LIQUID CHROMATOGRAM FOR CHRYSAZIN (1, 8 DIHYDROXYANTHRAQUINONE)

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Amendments Issued Since Publication

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